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Bis(3,5-dicarboxybenzoato-κ^2O,O')-(1,10-phenanthroline-κ^2N,N')-cadmium(II)

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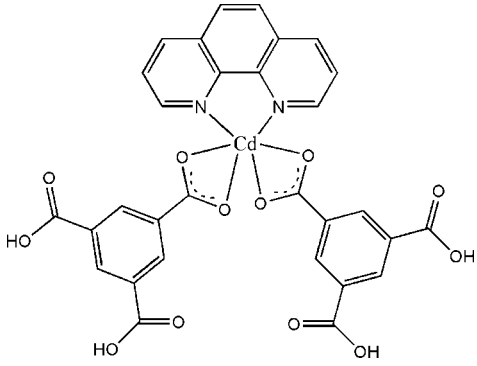
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.073; data-to-parameter ratio = 15.2.

The molecule of the title compound, $[Cd(C_9H_5O_6)_2(C_{12}H_8N_2)]$, has crystallographic twofold rotation symmetry. The Cd^{II} atom, located on the twofold axis, assumes a CdO_4N_2 distorted octahedral coordination geometry. In the crystal structure, the molecules link to each other by O—H···O and C—H···O hydrogen bonding to form a three-dimensional supramolecular network.

Related literature

For general background, see: Shi *et al.* (2004); Han *et al.* (2005).



Experimental

Crystal data

$[Cd(C_9H_5O_6)_2(C_{12}H_8N_2)]$
M_r = 710.86
Monoclinic, $C2/c$
a = 9.838 (2) Å
b = 16.541 (3) Å
c = 16.681 (3) Å
β = 96.32 (3)°
V = 2698.1 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.88 mm^{-1}
T = 298 (2) K
0.32 × 0.26 × 0.24 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.765, T_{max} = 0.816
5790 measured reflections
3095 independent reflections
2834 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.073
S = 1.05
3095 reflections
204 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cd1—N1	2.2671 (16)	Cd1—O2	2.3883 (15)
Cd1—O1	2.3079 (14)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O1^{i}	0.82	1.89	2.656 (2)	155
O5—H5A···O6^{ii}	0.82	1.81	2.623 (2)	170
C2—H2···O3^{i}	0.93	2.36	3.262 (3)	164

Symmetry codes: (i) $-x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $-x+1, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2344).

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supplementary materials

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Bis(3,5-dicarboxybenzoato-κ^2O,O')(1,10-phenanthroline-κ^2N,N')cadmium(II)

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Comment

As part of an ongoing investigation of metal complexes with the benzene-1,3,5-tricarboxylate ligand (Shi *et al.*, 2004; Han *et al.*, 2005), the structure of the title Cd^{II} complex is reported here. The asymmetric unit contains a half of Cd^{II} complex, with the Cd^{II} atom residing on a crystallographic twofold axis. The Cd^{II} ion has a distorted octahedral coordination geometry formed by four O atoms and two N atoms (Table 1). This arrangement appears to be the effect of the small bite angles produced by the chelating ligands. In the crystal the molecules are connected with O—H···O and C—H···O hydrogen bonding (Table 2), forming a three-dimensional supramolecular network.

Experimental

The compound was prepared by a hydrothermal method. A mixture of $Cd(NO_3)_2.4H_2O$ (0.5 mmol), benzene-1,3,5-tricarboxylic acid (0.6 mmol), 1,10-phenanthroline (0.6 mmol) and water (10 ml) was stirred for 20 min and then transferred to a 23 ml Teflon reactor. The reactor was kept at 433 K for 72 h under autogenous pressure. Single crystals were obtained after cooling to room temperature.

Refinement

H atoms were placed in calculated positions with C—H = 0.93 Å and O—H = 0.82 Å, and refined in riding mode with $U_{iso}(H) = 1.2U_{eq}(C)$ and $U_{iso}(H) = 1.5U_{eq}(O)$.

Figures

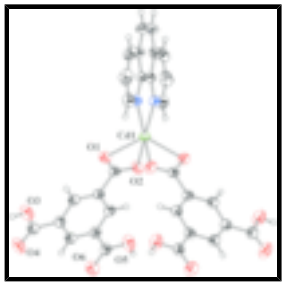

Fig. 1. The molecular structure of the title compound with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

Bis(3,5-dicarboxybenzoato-κ$^2O,O'$)(1,10-phenanthroline-κ$^2N,N'$)cadmium(II)

Crystal data

$[Cd(C_9H_5O_6)_2(C_{12}H_8N_2)]$ $F_{000} = 1424$

M_r = 710.86	D_x = 1.750 Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation λ = 0.71069 Å
Hall symbol: -C 2yc	Cell parameters from 5790 reflections
a = 9.838 (2) Å	θ = 2.4–27.5º
b = 16.541 (3) Å	μ = 0.88 mm^{-1}
c = 16.681 (3) Å	T = 298 (2) K
β = 96.32 (3)º	Block, colourless
V = 2698.1 (9) Å^3	0.32 × 0.26 × 0.24 mm
Z = 4	

Data collection

Bruker APEX CCD area-detector diffractometer	3095 independent reflections
Radiation source: fine-focus sealed tube	2834 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.033
T = 298(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −12→12
T_{min} = 0.765, T_{max} = 0.816	k = −21→21
5790 measured reflections	l = −21→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.030	H-atom parameters constrained
$wR(F^2)$ = 0.073	$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 1.679P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
3095 reflections	$\Delta\rho_{max}$ = 0.46 e Å^{-3}
204 parameters	$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-

factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.0000	0.095232 (10)	0.2500	0.03259 (8)
O1	−0.04641 (13)	0.16023 (9)	0.36635 (9)	0.0375 (3)
O2	0.15070 (14)	0.18897 (9)	0.32348 (10)	0.0413 (3)
O3	−0.22565 (16)	0.33500 (11)	0.56195 (12)	0.0573 (5)
H3A	−0.2837	0.3482	0.5911	0.086*
O4	−0.10746 (19)	0.43487 (13)	0.62596 (12)	0.0609 (5)
O5	0.42788 (16)	0.41835 (12)	0.44051 (11)	0.0528 (5)
H5A	0.4928	0.4496	0.4480	0.079*
O6	0.34627 (16)	0.49693 (11)	0.53307 (11)	0.0512 (4)
N1	0.13969 (15)	−0.01388 (10)	0.26674 (9)	0.0307 (3)
C1	0.07969 (18)	0.27267 (11)	0.42660 (11)	0.0292 (3)
C2	−0.02139 (18)	0.29186 (12)	0.47520 (12)	0.0327 (4)
H2	−0.0985	0.2593	0.4745	0.039*
C4	−0.00817 (18)	0.35978 (12)	0.52518 (12)	0.0328 (4)
C5	0.1072 (2)	0.40796 (12)	0.52686 (13)	0.0340 (4)
H5	0.1162	0.4534	0.5599	0.041*
C6	0.20969 (19)	0.38836 (12)	0.47902 (12)	0.0319 (4)
C7	0.19579 (18)	0.32127 (12)	0.42853 (12)	0.0312 (4)
H7	0.2639	0.3088	0.3961	0.037*
C8	0.06177 (17)	0.20275 (11)	0.36910 (11)	0.0296 (3)
C9	−0.1171 (2)	0.38168 (14)	0.57696 (14)	0.0395 (4)
C10	0.2741 (2)	−0.01298 (14)	0.28658 (13)	0.0409 (4)
H10	0.3193	0.0365	0.2907	0.049*
C11	0.3497 (2)	−0.08376 (17)	0.30141 (16)	0.0496 (6)
H11	0.4434	−0.0812	0.3169	0.060*
C12	0.2851 (2)	−0.15675 (15)	0.29300 (15)	0.0496 (6)
H12	0.3348	−0.2043	0.3020	0.060*
C13	0.1425 (2)	−0.15987 (12)	0.27059 (13)	0.0383 (4)
C14	0.07359 (18)	−0.08582 (10)	0.25986 (11)	0.0278 (3)
C15	0.0681 (3)	−0.23439 (13)	0.26011 (16)	0.0517 (6)
H15	0.1146	−0.2833	0.2674	0.062*
C17	0.33550 (19)	0.43895 (13)	0.48512 (12)	0.0346 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.04347 (13)	0.02012 (10)	0.03363 (12)	0.000	0.00178 (8)	0.000
O1	0.0322 (7)	0.0345 (7)	0.0470 (8)	−0.0104 (5)	0.0101 (6)	−0.0124 (6)
O2	0.0349 (7)	0.0409 (8)	0.0502 (9)	−0.0100 (6)	0.0148 (6)	−0.0157 (7)
O3	0.0398 (8)	0.0598 (10)	0.0774 (12)	−0.0229 (8)	0.0290 (8)	−0.0310 (9)
O4	0.0555 (10)	0.0641 (12)	0.0674 (12)	−0.0212 (9)	0.0270 (9)	−0.0347 (10)
O5	0.0361 (8)	0.0668 (11)	0.0580 (11)	−0.0252 (7)	0.0176 (7)	−0.0225 (8)

O6	0.0420 (8)	0.0523 (10)	0.0612 (10)	−0.0246 (7)	0.0142 (7)	−0.0223 (8)
N1	0.0301 (7)	0.0288 (7)	0.0329 (8)	−0.0011 (6)	0.0025 (6)	−0.0002 (6)
C1	0.0274 (8)	0.0265 (8)	0.0333 (9)	−0.0045 (6)	0.0011 (6)	−0.0018 (7)
C2	0.0291 (8)	0.0313 (9)	0.0378 (10)	−0.0098 (7)	0.0045 (7)	−0.0042 (7)
C4	0.0301 (8)	0.0329 (9)	0.0360 (10)	−0.0082 (7)	0.0067 (7)	−0.0053 (7)
C5	0.0325 (9)	0.0334 (10)	0.0364 (10)	−0.0110 (7)	0.0047 (7)	−0.0082 (7)
C6	0.0282 (9)	0.0334 (9)	0.0339 (9)	−0.0100 (7)	0.0024 (7)	−0.0021 (7)
C7	0.0261 (8)	0.0328 (9)	0.0345 (9)	−0.0053 (7)	0.0030 (6)	−0.0018 (7)
C8	0.0281 (8)	0.0261 (8)	0.0342 (9)	−0.0020 (6)	0.0016 (6)	−0.0006 (7)
C9	0.0368 (10)	0.0383 (10)	0.0455 (12)	−0.0117 (8)	0.0133 (8)	−0.0105 (9)
C10	0.0348 (10)	0.0482 (12)	0.0392 (11)	−0.0078 (8)	0.0025 (8)	−0.0022 (9)
C11	0.0318 (10)	0.0682 (16)	0.0482 (13)	0.0088 (10)	0.0018 (9)	−0.0036 (11)
C12	0.0462 (12)	0.0504 (14)	0.0509 (13)	0.0225 (10)	−0.0002 (9)	−0.0056 (10)
C13	0.0440 (11)	0.0317 (10)	0.0390 (11)	0.0110 (8)	0.0037 (8)	−0.0008 (8)
C14	0.0315 (9)	0.0260 (9)	0.0261 (9)	0.0015 (6)	0.0043 (6)	−0.0003 (6)
C15	0.0697 (15)	0.0252 (10)	0.0590 (15)	0.0095 (9)	0.0020 (11)	−0.0005 (9)
C17	0.0313 (9)	0.0375 (10)	0.0354 (10)	−0.0127 (8)	0.0055 (7)	−0.0045 (8)

Geometric parameters (Å, °)

Cd1—N1^{i}	2.2671 (16)	C2—C4	1.397 (3)
Cd1—N1	2.2671 (16)	C2—H2	0.9300
Cd1—O1	2.3079 (14)	C4—C5	1.385 (2)
Cd1—O1^{i}	2.3079 (14)	C4—C9	1.493 (3)
Cd1—O2	2.3883 (15)	C5—C6	1.391 (3)
Cd1—O2^{i}	2.3883 (15)	C5—H5	0.9300
Cd1—C8	2.6858 (19)	C6—C7	1.391 (3)
Cd1—C8^{i}	2.6858 (19)	C6—C17	1.488 (2)
O1—C8	1.272 (2)	C7—H7	0.9300
O2—C8	1.242 (2)	C10—C11	1.395 (3)
O3—C9	1.319 (2)	C10—H10	0.9300
O3—H3A	0.8200	C11—C12	1.364 (4)
O4—C9	1.198 (3)	C11—H11	0.9300
O5—C17	1.283 (3)	C12—C13	1.412 (3)
O5—H5A	0.8200	C12—H12	0.9300
O6—C17	1.246 (3)	C13—C14	1.402 (3)
N1—C10	1.327 (2)	C13—C15	1.434 (3)
N1—C14	1.355 (2)	C14—C14^{i}	1.449 (4)
C1—C2	1.387 (3)	C15—C15^{i}	1.345 (5)
C1—C7	1.394 (2)	C15—H15	0.9300
C1—C8	1.500 (2)		
N1^{i}—Cd1—N1	74.48 (8)	C5—C4—C9	119.20 (18)
N1^{i}—Cd1—O1	107.52 (5)	C2—C4—C9	120.98 (16)
N1—Cd1—O1	116.15 (6)	C4—C5—C6	119.90 (18)
N1^{i}—Cd1—O1^{i}	116.15 (6)	C4—C5—H5	120.0
N1—Cd1—O1^{i}	107.52 (5)	C6—C5—H5	120.0
O1—Cd1—O1^{i}	124.47 (8)	C7—C6—C5	120.28 (16)

$N1^{i}$—Cd1—O2	156.02 (6)	C7—C6—C17	121.04 (18)
N1—Cd1—O2	96.89 (6)	C5—C6—C17	118.65 (17)
O1—Cd1—O2	55.61 (5)	C6—C7—C1	119.98 (17)
$O1^{i}$—Cd1—O2	87.71 (6)	C6—C7—H7	120.0
$N1^{i}$—Cd1—$O2^{i}$	96.89 (6)	C1—C7—H7	120.0
N1—Cd1—$O2^{i}$	156.02 (6)	O2—C8—O1	121.27 (17)
O1—Cd1—$O2^{i}$	87.71 (6)	O2—C8—C1	119.47 (16)
$O1^{i}$—Cd1—$O2^{i}$	55.61 (5)	O1—C8—C1	119.22 (16)
O2—Cd1—$O2^{i}$	99.04 (8)	O2—C8—Cd1	62.77 (10)
$N1^{i}$—Cd1—C8	134.67 (6)	O1—C8—Cd1	59.14 (10)
N1—Cd1—C8	110.57 (6)	C1—C8—Cd1	169.85 (13)
O1—Cd1—C8	28.25 (5)	O4—C9—O3	124.2 (2)
$O1^{i}$—Cd1—C8	105.31 (6)	O4—C9—C4	124.38 (18)
O2—Cd1—C8	27.55 (5)	O3—C9—C4	111.46 (18)
$O2^{i}$—Cd1—C8	91.54 (6)	N1—C10—C11	122.1 (2)
$N1^{i}$—Cd1—$C8^{i}$	110.57 (6)	N1—C10—H10	118.9
N1—Cd1—$C8^{i}$	134.67 (6)	C11—C10—H10	118.9
O1—Cd1—$C8^{i}$	105.31 (6)	C12—C11—C10	119.4 (2)
$O1^{i}$—Cd1—$C8^{i}$	28.25 (5)	C12—C11—H11	120.3
O2—Cd1—$C8^{i}$	91.54 (6)	C10—C11—H11	120.3
$O2^{i}$—Cd1—$C8^{i}$	27.55 (5)	C11—C12—C13	119.8 (2)
C8—Cd1—$C8^{i}$	97.07 (8)	C11—C12—H12	120.1
C8—O1—Cd1	92.62 (11)	C13—C12—H12	120.1
C8—O2—Cd1	89.68 (11)	C14—C13—C12	117.01 (19)
C9—O3—H3A	109.5	C14—C13—C15	120.15 (19)
C17—O5—H5A	109.5	C12—C13—C15	122.83 (19)
C10—N1—C14	119.13 (17)	N1—C14—C13	122.39 (17)
C10—N1—Cd1	126.54 (14)	N1—C14—$C14^{i}$	118.52 (10)
C14—N1—Cd1	114.20 (11)	C13—C14—$C14^{i}$	119.08 (12)
C2—C1—C7	119.52 (17)	$C15^{i}$—C15—C13	120.75 (12)
C2—C1—C8	120.57 (16)	$C15^{i}$—C15—H15	119.6
C7—C1—C8	119.83 (17)	C13—C15—H15	119.6
C1—C2—C4	120.49 (16)	O6—C17—O5	124.15 (17)
C1—C2—H2	119.8	O6—C17—C6	119.21 (18)
C4—C2—H2	119.8	O5—C17—C6	116.62 (18)
C5—C4—C2	119.82 (17)		

Symmetry codes: (i) $-x, y, -z+1/2$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···$O1^{ii}$	0.82	1.89	2.656 (2)	155
O5—H5A···$O6^{iii}$	0.82	1.81	2.623 (2)	170
C2—H2···$O3^{ii}$	0.93	2.36	3.262 (3)	164

Symmetry codes: (ii) −*x*−1/2, −*y*+1/2, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*+1.

Fig. 1